FEBRUARY, 1909 Chemical Series Vol. I, No. 7

Biological & Medical Serials

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Memoirs of the Department of Agriculture in India

STORAGE

THE SYSTEM WATER, CALCIUM CARBONATE, CARBONIC ACID

BY

J. WALTER LEATHER, Ph.D., F.I.C., F.C.S

Imperial Agricultural Chemist

AND

JATINDRANATH SEN, M.A., F.C.S

Assistant to the Imperial Agricultural Chemist

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AGRICULTURAL RESEARCH INSTITUTE, PUSA

PUBLISHED FOR

THE IMPERIAL DEPARTMENT OF AGRICULTURE IN INDIA

BY

THACKER, SPINK & CO., CALCUTTA

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PREFACE.

In the course of some investigations on soil-gases, and the available plant food in soils, which are in progress in the Chemical Section of the Agricultural Research Institute at Pusa, more accurate information was required on the concentration of calcium carbonate and carbonic acid in water than exists in the literature of this subject. Accordingly, a series of measurements have been made, and the data, together with the general formula expressive of the concentration, are published in this memoir.

I desire to take the opportunity of expressing my obligation to Dr. Morris W. Travers, F.R.S., for kindly advice in regard to the interpretation of some of the results.

J. W. L.

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PLATE I.

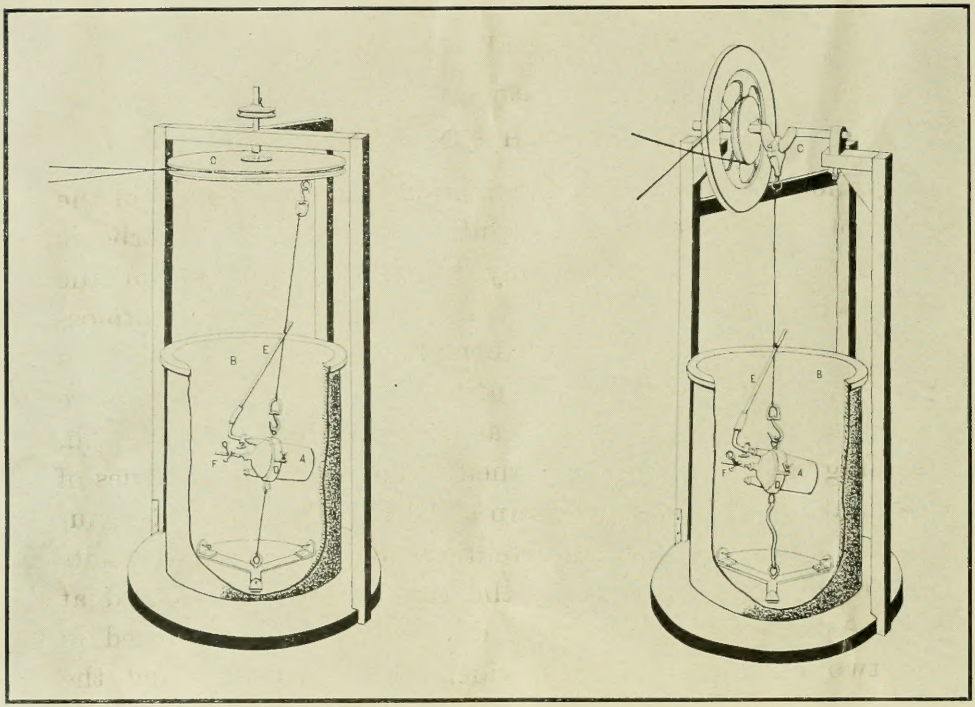

Fig. 1.

Fig. 2.

THE SYSTEM WATER, CALCIUM CARBONATE, CARBONIC ACID.

BY

J. WALTER LEATHER, Ph.D., F.I.C., F.C.S.;

AND

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ALTHOUGH numerous determinations have been made of the amount of calcium carbonate in solution in presence of carbonic acid, the literature contains only two communications on the subject which attempt to show the relation obtaining at atmospheric pressure between the concentration of the calcium carbonate, or bicarbonate, in solution and the partial pressure of the carbon dioxide in the gas phase. The one is by Th. Schloesing who in 1872 published* the results of a series of determinations on this relationship at 16°C. The other communication is by Treadwell and Reuter who published† the results of a series of determinations on the same subject, conducted at 15°C. Apart from the fact that the concentrations obtained in these two investigations differ widely from each other, and the uncertainty which consequently existed as to which was the more reliable, we required correct information for temperatures up to about 40°C. for the assistance of the other work which has been briefly referred to in the Preface.

It is of interest to refer to the methods employed in each of these investigations, because the accuracy of the data must depend in a great measure on this. It is obvious that whether carbonic acid is brought into contact with calcium carbonate

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* *Compt. Rend.*, 74 (1872), 1552; and *Jour. Chem. Soc.*, 25 (1872), 788.

† *Zeits.* f. *anorg. Chemie*, XVII (1898), p. 170 et seq.

and water, or conversely whether a solution of "calcium bicarbonate" is exposed to an inactive gas, the reaction is a slow one, and equilibrium will only be attained in a comparatively brief period if the contact between the several phases is intimate. Schloesing pumped air containing known amounts of carbon dioxide through water containing calcium carbonate in excess for six or seven days at constant temperature (16°C), when he considered that equilibrium had set in. He says "Quand l'équilibre est établi dans la dissolution, e'est-à-dire après six ou sept jours,....." (p. 1554), but does not explain how he ascertained that equilibrium was established. Unless equilibrium had actually occurred, his concentration would tend to be too low in comparison with the partial pressure of the CO_2. Schloesing considered that the solution consisted of calcium carbonate of the same concentration as pure water would contain at 16°C., carbonic acid of the same concentration as pure water would contain at 16°C. and under the partial pressure of the system, and calcium bicarbonate of the concentration which is demanded by the formula*

$$x^m = ky$$

Where x = the partial pressure of the gaseous CO_2 and m = ·37866.

A comparison between our data and those of Schloesing may be here suitably made in so far as this is possible.

Partial pressure of CO_2 in gas.	Schloesing. t = 16°C.		Leather and Sen. t = 15°C.	
P.	$CaCO_3$.	excess CO_2.	$CaCO_3$.	excess CO_2.
	milligrammes per 100 c.c.			
1·4	22·3	12	...	...
1·5	...	...	19·3	15 2
1·7	...	...	23·8	13 5
5·0	36·0	25·0	...	...
6·8	...	...	44·5	32·7
13·6	...	...	72·3	56·0
25·4	66·3	78·1	...	...
31·6	...	...	105·0	111·7
41·7	78·7	115·4	...	...

* *Compt. Rend.*, 75 (1872), 70 ; and *Jour. Chem. Soc.*, 25 (1872), 880.

The differences at low concentrations are only small, but at higher concentrations, ours are considerably greater than Schloesing's.

Treadwell and Reuter exposed a solution of calcium carbonate in carbonic acid to air in large bottles, which were *stationary*, and considered that the system had attained equilibrium when no further change in the composition of the gas phase was noticed. The vessels had a capacity of about 20 litres and were about half full of liquid. Thus they adopted the slowest possible method devisable for the end in view; for although the solution near the surface of the liquid might be expected to approach a state of equilibrium with the gas fairly quickly, the "bicarbonate" in the remainder of the liquid could only arrive at the surface by diffusion. As a result, their data for the concentration might be expected to be too high in comparison with the partial pressure of the CO_2. In fact, they seem to have recognised this defect, for they say "Auch hier ist in erster Linie das Erhalten einer Durchschnittsprobe von Wichtigkeit, denn es ist klar, dass das Wasser nicht in jedem Hohenschicht gleichmässig seine absorbierte Kohlensäure abgiebt; am meisten Gas verliert die oberste, am wenigsten die unterste Schicht" (p. 173). This necessarily implies an admission that the solution was not in equilibrium and consequently detracts much from the reliability of the method. The following comparison may be made between the concentrations as found by these authors and ourselves.

Partial pressure of CO_2 in gas. P.	Treadwell and Reuter. t = 15°C.		Leather and Sen. t = 15°C.	
	$CaCO_3$.	excess CO_2.	$CaCO_3$.	excess CO_2.
	milligrammes per 100 c.c.			
·9	77·1	48·4	—	—
1·7	—	—	23·8	13·5
2·0	82·2	60·5	—	—
6·8	108 3	134·0	44·5	32·7
9·9	—	—	62·7	45·6
10·0	115·6	207·8	—	—

This comparison sufficiently exhibits the great divergence between our results and those of Treadwell and Reuter and

illustrates the necessity of providing for very intimate contact between the two phases in all such work.

It was early recognised by us that the greatest care must be adopted in order to obtain even approximate equilibrium. Schloesing provides only a verbal explanation of his apparatus in his communication, and in our hands at least the aspiration of carbon dioxide and air, through water containing calcium carbonate in suspension, showed that the contact was very imperfect and equilibrium could not be obtained in any short time. On the other hand, if an aqueous solution of calcium carbonate in carbonic acid be *agitated* with air, the reaction proceeds rapidly and equilibrium sets in in the course of two or three hours. The reverse change, namely, the solution of calcium carbonate, when shaken with water and air containing carbon dioxide, proceeds materially more slowly, and must also depend on the fineness of the carbonate ; if it is freshly precipitated, equilibrium sets in in the course of three or four hours, but if the particles are larger, the process is more protracted ; simple boiling of the precipitate is sufficient to make a material difference in this respect. Had the necessary apparatus been at our disposal, we would have preferred to employ both reactions, but practically our experiments had to be completed during the working day, and consequently, although a number of determinations have been made from $CaCO_3 + CO_2$, we have depended on the results of those which have proceeded from the solution + air, because equilibrium could be attained so much more quickly.

Methods of analysis used.—Before describing the special apparatus which has been employed for the investigation, it will be convenient to mention what analytical methods were used. The carbon dioxide in the gas phase was determined gasometrically and the concentration expressed as a percentage ; the water vapour was naturally included as a part of the gas. The calcium was determined by titration of the solution, after filtering off the insoluble carbonate, with dilute acid, methyl orange being used as indicator. The "free" carbonic acid was determined by boiling the (unfiltered) liquid and absorbing in soda lime.

The method of obtaining equilibrium between the several components.—After some preliminary work, a number of experiments were made with a vessel which contained the solution, excess of calcium carbonate and air. This vessel was provided with a tap by means of which (*inter alia*) momentary communication could be made with the atmosphere. It was held in a water bath of constant temperature. Periodically (every 10 to 15 mins.) it was taken out and the liquid agitated. Some time after no further alteration of pressure could be perceived, the vessel was attached to a gas burette, and while a sample of the gas was withdrawn, mercury was allowed to flow in, thus preventing any serious alteration of pressure. Then, samples of the solution were withdrawn for the determination of the calcium carbonate and carbonic acid. We considered the apparatus defective simply because intimate contact between gas and liquid was only ensured during the brief period of agitation. A second apparatus which we devised is shown in plate No. I, fig. 1, in which a Woulff's bottle A served as the reaction vessel. The horizontal pulley C revolved and carried the Woulff's bottle in a circular path, but since the latter was prevented from revolving, the liquid was dashed against the end of the bottle at each revolution of the pulley.

Fig. 2 in the plate illustrates the apparatus with which we made all the later determinations. A Woulff's bottle A again serves as the reaction vessel, but in place of a circular motion, the latter is vertical ; the clamp D holding the bottle is attached by an India-rubber cord to the bottom of the water bath B ; when the crank C is turned down, the rubber is slack ; when it becomes horizontal, the rubber is just tight, and naturally becomes stretched while the crank moves through the upper semi-circle. Consequently the bottle receives a jerk at every revolution of the pulley. The bottle is provided with a long capillary tube E and stop cock which admits of re-establishment of normal pressure by communication with the outside atmosphere. This stop cock was opened frequently at first and somewhat less often during the later stages of the reaction. Equilibrium was assumed to have set in when no change of pressure could be perceived on opening

this stop cock ; the agitation was, however, continued for some two hours longer in order to secure as complete a change as possible. After this the bottle was removed, attached to a gas burette, mercury run in at F and gas withdrawn at E. The samples of solution were then immediately taken for estimation of dissolved carbonic acid and calcium oxide. The calcium oxide employed for the solution of "bicarbonate" was obtained by burning a sample of very good marble.

The solid phase consists of $CaCO_3$ only.—In order to determine whether the solid phase in the system $CaCO_3$, CO_2, water contained any bicarbonate, two experiments were made. The separation of the solid for direct examination without decomposition of any bicarbonate (if such existed) appeared to us impossible, and recourse was consequently had to the following indirect method. A solution of calcium carbonate in carbonic acid was prepared and then agitated with air so as to produce equilibrium. Two portions of the liquid were then boiled and the carbon dioxide, which was evolved, determined. The one portion included the precipitated carbonate ; the other was filtered rapidly into the boiling flask. The results are as follows :—

	CO_2 evolved from 100 c.c. solution— without filtering off solid.	after removal of solid.
	grms.	grms.
I	·1261	·1211
II	·0837	·0760

In each case slightly less CO_2 was obtained after separation of the precipitate, which might be urged as evidence that the precipitate contains some small amount of bicarbonate, but we consider such a conclusion unwarranted. We had no means of executing the filtration in an atmosphere of the same concentration of carbon dioxide gas as had been in equilibrium with the solution, and were compelled to expose the liquid to the laboratory atmosphere. Such exposure would necessarily cause a certain amount of the dissolved carbonic acid to pass into the air, and it was anticipated that less carbonic acid would be found in the filtered than in the unfiltered solution. Since the difference

between the two determinations is so small, we consider the absence of the bicarbonate from the solid phase is proved.

Whilst writing this memoir we have seen the records of two series of experiments which have been made to ascertain the existence of solid calcium bicarbonate. In the first of these Cameron and Robinson* determined the relation between pressure and concentration of carbonic acid in the presence of calcium carbonate and water at 0°C. up to 4·5 atmospheres, and found that the one increased with the other. Had a bicarbonate formed, the pressure would have remained constant during its formation, and they accordingly conclude that no solid bicarbonate is formed. In the second series of experiments Keiser and McMaster† precipitated a solution of calcium chloride with ammonium bicarbonate, both solutions being first reduced to 0°C. The precipitate was rapidly filtered and washed without any appreciable rise of temperature being permitted ; this precipitate was then found to have the formula $CaCO_3$ 1·75 H_2CO_3. They also examined the solution which had been first agitated with air, and found that the relation between the calcium carbonate and carbonic acid to be the same as in the solid. Since the temperature 0°C is so much below what we have employed in our experiments, it is unsafe to criticise this work on the basis of our own, but judging from the concentrations of the two constituents in solution as found by Keiser and McMaster, it seems likely that these solutions were far from being in equilibrium with the air, and that had they been brought into equilibrium, not only would the concentrations have fallen very considerably, but the ratio between $CaCO_3$ and H_2CO_3 would have altered materially. The presence of the carbonic acid in the precipitate is not so readily accounted for, but it seems reasonable to suggest that it was present as a solid solution rather than as a part of a definite chemical compound. It seems to us at least proved that at the temperature 15°C. and upwards calcium bicarbonate cannot exist in the solid state.

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* *Jour. Phys. Chem.*, 1908, XII, 561.

† *Jour. Am. Chem. Soc.*, 1908, XXX, 1714.

Mode of expressing the results.—Apart from any consideration of the existence or otherwise of the bicarbonate, it is much simpler for all practical purposes to consider the components of the solution to be calcium carbonate and carbonic acid respectively and to calculate each as a function of the partial pressure.

Therefore, from the data obtained at each temperature the constants in the expression

$$C^n = kp$$

have been evaluated for $CaCO_3$ and CO_2 respectively.

On plotting the values of these *constants* against the temperature, they were found to be slightly irregular as was indeed anticipated, but as they showed no indication of lying on curves, it has been assumed that they are linear functions of the temperature, and that the value of n may be expressed as $\alpha + \beta t$, that of log k as $\gamma + \delta t$. Hence the concentration of either constituent may be expressed thus :—

$$\log C_{(CaCO_3)} = \frac{(\gamma + \delta t) + \log p}{\alpha + \beta t}$$

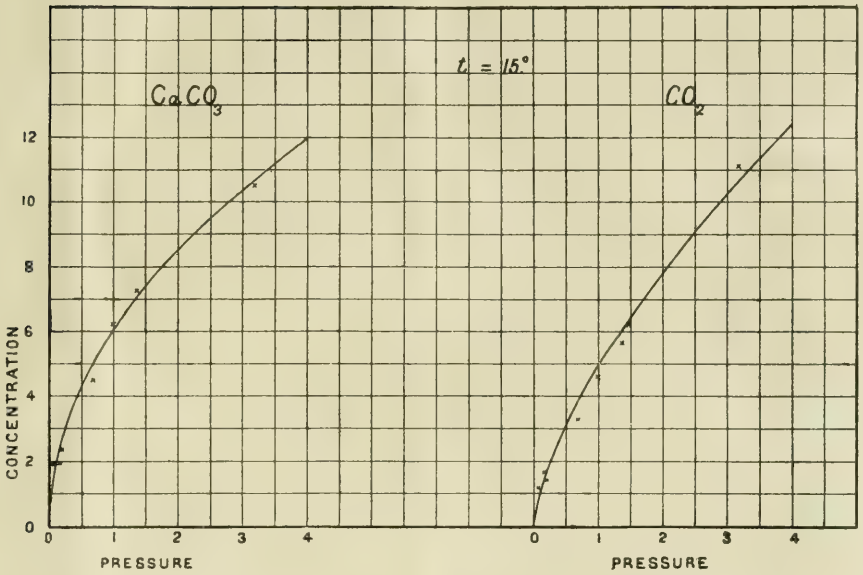
$$\log C_{(CO_2)} = \frac{(\gamma + \delta t) + \log p}{\alpha + \beta t}$$

It is naturally understood that the values of α, β, γ and δ are different in the two cases. Their values are as follows :—

	for $CaCO_3$.	for CO_2.
α	1·5897	1 2133
β	0310	·0200
γ	2·2853	1·4213
δ	·0256	·0096

In the following pages are set out for each temperature the experimental data, and those calculated from the general formulæ, in charts and tabular statements.

PLATE II.



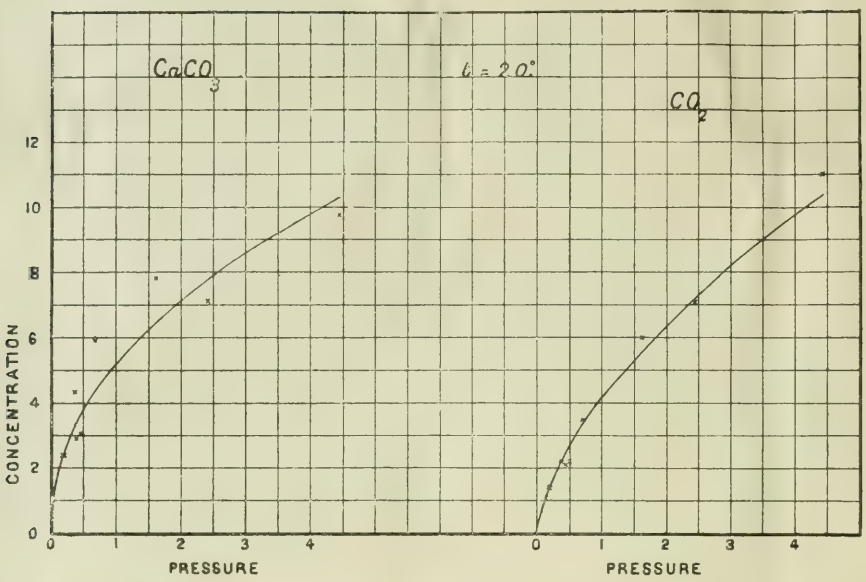
TEMPERATURE 15°C.

The subjoined statement shows the concentration of $CaCO_3$ and CO_2 *in m.grms. per* 100 *c.c.* as experimentally found and as calculated from the general formula.

For $CaCO_3$, $n = 2{\cdot}0554$, $\log k = 2{\cdot}6693$.
For CO_2, $n = 1{\cdot}5127$, $\log k = 1{\cdot}5659$.

P.	$CaCO_3$.		CO_2.	
	Found.	Calculated.	Found.	Calculated.
·8	19·3	17·9	11·7	9·35
1·5	19·3	24·2	15·2	14·2
1·7	23·8	25·8	13·5	15·4
6·8	44·5	50·6	32·7	38·5
9·9	62·7	60·8	45·6	49·2
13·6	72·3	71·0	56·0	60·8
14·6	68·6	73·5	62·3	63·7
31·6	105·0	107·0	111·7	106·2

PLATE III.



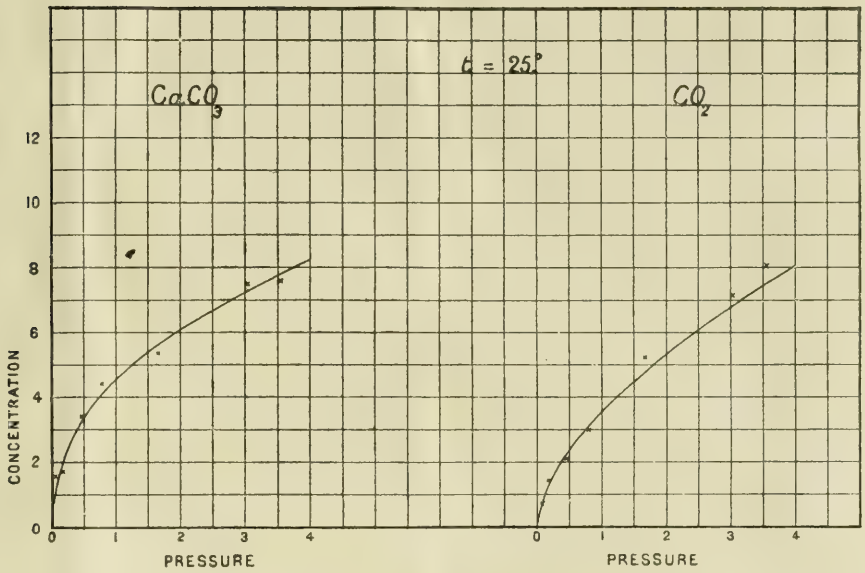
Temperature 20°C.

The subjoined statement shows the concentration of $CaCO_3$ and CO_2 *in m.grms. per* 100 *c.c.* as experimentally found and as calculated from the general formula.

For $CaCO_3$, n = 2·2107, log k = 2·7973.
For CO_2, n = 1·6125, log k = 1·6141.

P.	$CaCO_3$.		CO_2.	
	Found.	Calculated.	Found.	Calculated.
1·8	23·9	24·0	14·9	14·4
3·6	43·2	32·9	22·2	22·2
3·9	29·1	34·1	21·7	23·3
4·8	30·2	37·4	22·2	26·5
6·9	58·4	44·2	34·6	33·2
16·0	78·0	64·6	59·9	56·0
24·1	70·9	77·7	70·4	72·1
41·3	97·9	102·4	110·0	105·2

PLATE IV.



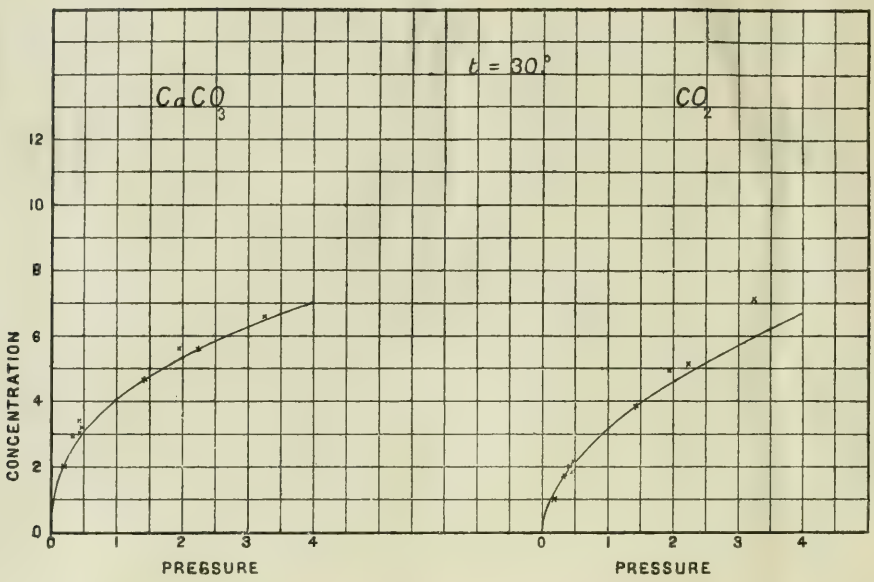
TEMPERATURE 25°C.

The subjoined statement shows the concentration of $CaCO_3$ and CO_2 *in m.grms. per* 100 *c.c.* as experimentally found and as calculated from the general formula.

For $CaCO_3$, $n = 2{\cdot}3659$, $\log k = 2{\cdot}9253$.
For CO_2, $n = 1{\cdot}7124$, $\log k = 1{\cdot}6623$.

P.	$CaCO_3$.		CO_2.	
	Found.	Calculated.	Found.	Calculated.
·7	15·9	14·8	9·1	7·6
1·6	17·7	21·0	11·1	12·3
4·6	34·1	32·9	20·8	22·8
7·8	44·6	41·1	30·1	31·0
16·5	53·9	56·4	52·2	48·1
30·1	74·3	72·6	71·5	68·2
55·5	75·5	78·0	80·3	75·2

PLATE V.



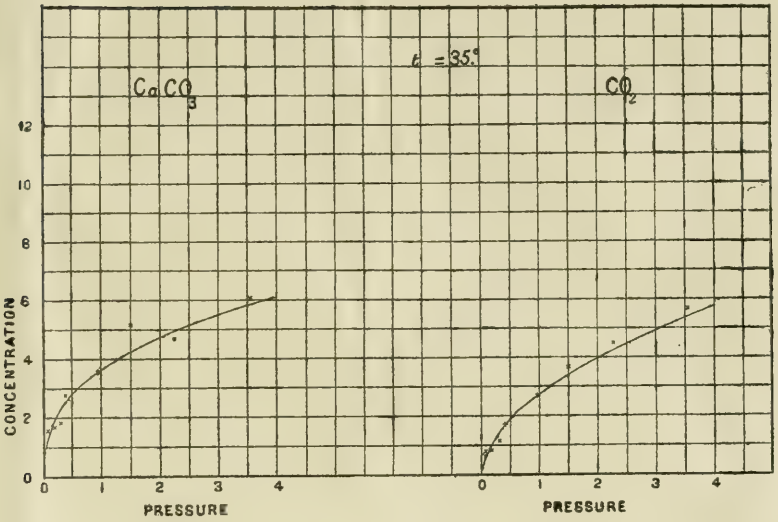
TEMPERATURE 30°C.

The subjoined statement shows the concentration of $CaCO_3$ and CO_2 *in m.grms. per* 100 *c.c.* as experimentally found and as calculated from the general formula.

For $CaCO_3$, $n = 2{\cdot}5211$, $\log k = 3{\cdot}0533$.
For CO_2, $n = 1{\cdot}8122$, $\log k = 1{\cdot}7104$.

P.	$CaCO_3$.		CO_2.	
	Found.	Calculated.	Found.	Calculated.
1·8	20·0	20·5	10·1	12·2
3·3	29·5	26·1	16·9	17·0
4·2	33·9	28·7	19·9	19·4
4·4	30·2	29·2	18·5	19·9
4·7	31·6	30·1	21·0	20·7
14·1	47·1	47·3	38·5	38·8
19·6	56·1	53·0	49·6	45·4
22·2	55·9	55·6	51·4	48·6
32·2	65·5	64·4	71·0	59·7

PLATE VI.



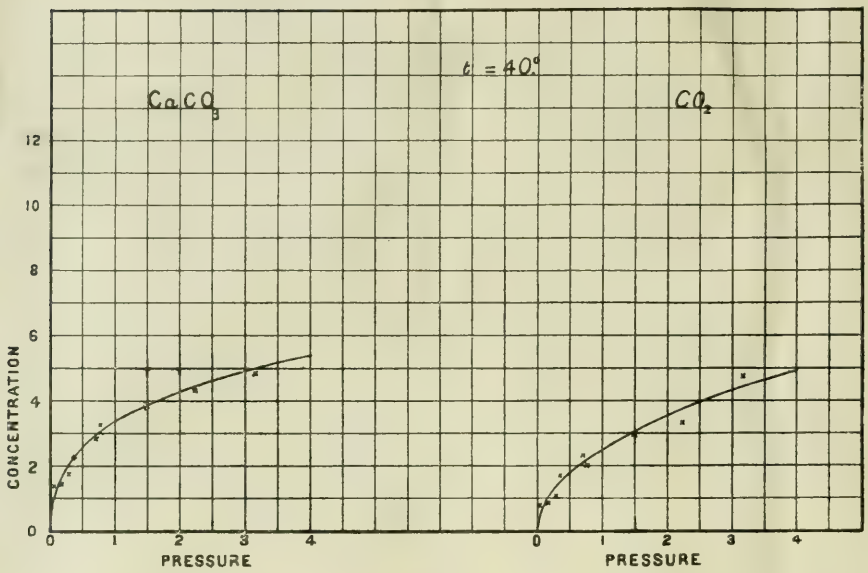
Temperature 35°C.

The subjoined statement shows the concentration of $CaCO_3$ and CO_2 *in m.grms. per* 100 *c.c.* as experimentally found and as calculated from the general formula.

For $CaCO_3$, n = 2·6764, log k = 3·1813.
For CO_2, n = 1·9120, log k = 1·7586.

P.	$CaCO_3$.		CO_2.	
	Found.	Calculated.	Found.	Calculated.
·7	15·7	13·5	9·0	6·9
1·6	16·8	18·4	9·3	10·6
2·9	18·2	23·0	11·3	14·5
4·0	27·5	25·9	17·8	17·2
9·6	35·7	36·0	26·9	27·2
15·0	51·6	42·5	37·6	34·3
22·6	46·4	49·6	45·2	42·5
35·4	60·0	58·5	56·8	53·7

PLATE VII.



TEMPERATURE 40°C.

The subjoined statement shows the concentration of $CaCO_3$ and CO_2 *in m.grms. per* 100 *c.c.* as experimentally found and as calculated from the general formula.

For $CaCO_3$, $n = 2{\cdot}8316$, $\log k = 3{\cdot}3093$.
For CO_2, $n = 2{\cdot}0118$, $\log k = 1{\cdot}8068$.

P.	$CaCO_3$.		CO_2.	
	Found	Calculated.	Found.	Calculated.
·6	13·6	12·3	7·8	6·1
1·7	14·3	17·8	8·5	10·3
2·9	17·5	21·5	10·6	13·4
3·5	23·2	23·0	16·9	14·8
7·0	28·4	29·3	23·4	20·8
7·7	32·5	30·3	20·0	21·8
14·9	38·4	38·3	29·3	30·3
22·2	42·7	44·1	33·3	36·9
31·7	48·0	50·0	47·6	44·1

Conclusion.

The information contained in this Memoir on the solubility of calcium carbonate in the presence of carbonic acid is obviously a step towards the possession of more exact knowledge than we at present possess regarding the concentration of lime in the aqueous solution in the soil. Although impossible to obtain this solution from the soil in an unaltered state, it is feasible to obtain specimens of the gases, and from a knowledge of their composition to make deductions regarding the amount of carbonic acid in solution and hence also regarding the concentration of calcium and other carbonates. Our prospective work naturally includes a study of the relations subsisting between the carbonic acid and other carbonates commonly present in the soil, in regard to which such information may be lacking. The conclusion of this work should place a general formula in our hands with the aid of which our object may be attained.

Although only one step has been achieved, it has been possible to already apply it towards a study of the solubility of soil phosphates in the presence of calcium carbonate and carbonic acid. The solubility of soil and rock minerals has been repeatedly shown to be much greater in the presence of carbonic acid than in pure water, and accordingly this constituent has been considered to be an important natural solvent of plant food in the soil. Such experiments have been, however, uniformly made with pure carbonic acid in such concentration as can rarely if ever occur in arable land. On the other hand if soils are treated with water, calcium carbonate and carbonic acid in such proportions as occur naturally, the result may be a quite different one. For example, a number of soils have been placed under these circumstances and in no case was it found that the solubility of the phosphate was increased; generally indeed the effect was a somewhat reduced solubility.

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